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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re: Application of SHEMBEL, et al.

Application No. 10/038,556

Examiner: Dove, Tracy M.

Date Filed: January 4, 2002

Group: 1745

For: SOLID POLYMER ELECTROLYTE LITHIUM BATTERY

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I, Yurii Maletin, PhD and DSc, declare as follows:

1. I am familiar with patent application No. 10/038,556 entitled " SOLID
POLYMER ELECTROLYTE LITHIUM BATTERY" (hereafter the '556 application) and the
subject matter described therein.

{WP269192;1}

2. As detailed in the attached cv, I hold a PhD degree at the Institute of General and Inorganic Chemistry, Kiev, 1977 and Dr. of Science degree at the Institute of Chemical Physics, Moscow, 1989 and have worked regularly organic electrolytes to be used in various energy storage devices including lithium chemical power sources for over ca. 20 years. I am presently working as a consultant for Ener1, Inc.

3. I have authored or coauthored more than 15 scientific papers, and more than 10 issued patents regarding electrolytes for chemical power sources including lithium ones.

4. I have reviewed the Office Action dated May 24, 2004 and references cited therein, including U.S. Patent No. 6,617,078 to Chia et al. ("Chia") and U.S. Patent No. 5,389,463 to Chang et al ("Chang"). Chia discloses production of a gel electrolyte in two steps. A micro porous film separator is formed by mixing of chlorinated PVC and terpolymer of vinylidene chloride. The solid polymer separator is impregnated with a 1M solution of LiPF_6 in ethylene carbonate and dimethyl carbonate-based electrolytes. This conventional process clearly produces a two phase gel electrolyte, where the electrolyte fills the pores of the polymer. Chang only discloses a porous separator. To produce an electrolyte for use in a battery, the porous separator would be impregnated with a liquid electrolyte, where the electrolyte would become disposed in the pores of the separator. The electrolyte would then provide Li ion conductivity. As a result, Chang's hypothetical impregnated separator would consist of two separate phases analogous to Chia (discussed above) with the first phase being a solid porous separator, and the second phase being a liquid electrolyte including a salt.

5. The polymer electrolyte described in the '556 application is a polymer electrolyte comprising a modified chlorine containing polymer having an enhanced chlorine level (e.g. C-PVC), relative to the chlorine content of an unmodified chlorine containing polymer formed from polymerization of its monomer. The polymer electrolyte is a homogeneous single phase material that includes the modified polymer, a salt of an alkali metal and an aprotic solvent, where all the electrolyte components are uniformly distributed in the electrolyte and can be properly regarded as being a solid electrolyte, despite the fact that the solvent is a liquid at room temperature conditions. Some solid polymer electrolytes having aprotic solvents therein were known prior to the filing date of the above-referenced application. Such homogeneous single phase systems including electrolytes according to the invention are generally made possible by strong intermolecular interactions between polymer and solvent molecules under certain conditions, such as the dual solvent process disclosed in the '556 application where the polymer electrolyte goes into solution with the other electrolyte components.

Specifically, as described in the '556 application, such as page 21, lines 11-15, and Examples 2-6, a low boiling point solvent is used to first dissolve the salt, aprotic solvent (plasticizer) and the modified polymer. Thus, at least two solvents are used. The low boiling point solvent is then removed in a drying process, preferably under vacuum. The concentration of the various polymer electrolyte components and the drying conditions as described in the '556 application essentially eliminate any phase separation as the homogeneous solution is dried to form a single phase homogeneous polymer electrolyte claimed in the '556 application.

The polymer electrolyte of the claimed invention formed using the homogeneous process described therein inherently provides a single phase homogeneous polymer comprising material comprising the polymer, the salt and the aprotic solvent, provided the salt loading level is not too

high. The salt levels disclosed in the application do not appear high enough to cause phase separation. Such a homogeneous single phase solid system can be formed due to strong intermolecular interactions between polymer and solvent molecules under conditions disclosed in the present application. The single phase result would be clear to one having ordinary skill in the art based on the application.

6. I have reviewed the comparative AFM data provided with Dr. Shembel's Declaration filed in this case. Such data clearly demonstrates the structural differences between the claimed single phase polymer electrolyte and the gel electrolytes disclosed in the Chia and Chang. Based on Dr. Shembel's sworn testimony the structure analyzed in Fig. 1 was a single phase electrolyte according to the invention formed from a lithium salt (LiCF_3SO_3) solution in an aprotic solvent (PC) which was added into previously prepared C-PVC solution in tetrahydrofuran (THF). Solutions, prepared in this fashion, were poured onto a glass support and dried, first at room temperature for 24 h and then in a vacuum at 45°C for 48 h. These steps follow the method described in the '556 application. The resulting electrolyte films are single phase homogeneous systems that have a uniform distribution of the all components in electrolyte as evidenced by the very flat electrolyte surface shown in Fig. 1.

7. The method of electrolyte preparation described in Chang and Chia include the steps of production of microporous solid separator based on C-PVC formed by drying from polymer solution in tetrahydrofuran, impregnation of the produced separator with a liquid electrolyte, which is 0.5M solution of LiCF_3SO_3 in propylene carbonate, and followed by drying

the produced polymer electrolyte at room temperature for 24 h and then in a vacuum at 45°C for 48 h.

The films produced by the method of Chang and Chia were found to have a rough surface evidenced by the AFM shown in Fig. 2. The respective electrolyte components are distributed non-uniformly (Fig. 2), and where individual crystallites of the LiCF_3SO_3 salt exited the surface of the electrolyte.

8. Comparative analysis of the AFM polymer electrolytes based on C-PVC shown above demonstrates that homogeneous single phase electrolytes produced from drying homogeneous solutions according to the '556 application is clearly structurally distinguishable as compared to the multi-phase gel electrolytes formed using separator impregnation with liquid electrolytes as described in Chang and Chia.

9. I cannot agree with the examiner's assertion that it is obvious to substitute the chlorinated PVC of Chang or Chia with Alamgir's electrolyte to arrive at the claimed invention in the present application. The change in polymer chemical structure and composition can essentially change its interaction with other molecules, e.g., solvents and salts. And as noted above, this is the intermolecular interaction that enables to obtain a single phase solid electrolyte. Such homogeneous electrolytes may have different and/or limited stability in different electrolytes, and the result now claimed by Applicants can hardly be obvious *a priori*.

10. I further state that all statements made herein are of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that

these statements were made with my knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under §1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.



Dr. Yuri Maletin

Nov 24, 2005

Date

Curriculum Vitae

Name	Yurii A. Maletin
Born	January 15, 1949
Place of birth	Moscow, Russia
Graduated from	Moscow State University after M.V. Lomonosov, Chemical Department, MSc in Chemistry, 1971
Scientific Degrees	PhD in Inorganic Chemistry, supported at the Institute of General and Inorganic Chemistry, Kiev, 1977 DSc in Physical Chemistry, supported at the Institute of Chemical Physics, Moscow, 1989
Most recent positions	Head of the Physical Chemistry Department, National Technical University of Ukraine "KPI", Kiev, starting from 2002 Head of the Coordination Chemistry Department, Institute of General and Inorganic Chemistry, National Academy of Science of Ukraine, Kiev, 1987-2002
Scientific activities	Fellow of the Royal Society of Chemistry (UK), starting from 1996 Member of the Advisory Board of the Inorganic Chemistry Communications, 1998-2002 Member of All-Ukrainian Board on Inorganic Chemistry, starting from 1978 Member of the Supreme Certificate Commission of Ukraine to award scientific degrees in Chemistry, starting from 2004
Publications	64 scientific papers in referred journals and 19 patents
Most recent international presentations	2003 Int. Conference on Advanced Capacitors, Kyoto, Japan, May, 2003. NATO Carbon Advanced Research Workshop and Conference, Argonne National Laboratory, USA, Oct. 2003. Advanced Capacitor World Summit, Washington, DC, July, 2004 14 th Int. Seminar on Double Layer Capacitors and Similar Energy Storage Devices, Deerfield Beach, Florida, Dec. 2004.
Date:	December 27, 2004
Signature:	

(WP269194:11)

Yurii Maletin: List of publications

over the past 25 years after PhD thesis:

(NOTE: a great part of information on supercapacitors is confidential so the number of publications in that area is mostly limited by those that have been included in Proceedings of various international conferences)

1. Y.A. Maletin, N.G. Strizhakova, and I.A. Sheka. Effect of metal ions on the electron transfer between organic radicals. *Doklady AN SSSR (Russ.)*, 1979, **248**, No.3, pp. 651-654.
2. N.G. Strizhakova, Y.A. Maletin, and I.A. Sheka. Interaction of Cu^{2+} compounds with triphenyl verdazyl radical. *Ukrain. J. Chem. (Russ.)*, 1979, **45**, No. 11, pp. 1039-1041.
3. Y.A. Maletin, and I.A. Sheka. 3d-Metal complexes with 1,2,4-trioxo-3,4,5-tris (methoxy carbonyl) cyclopentadienone. *Coord. Chim. (Russ.)*, 1980, **6**, No.1, pp. 81-85.
4. Y.A. Maletin, N.G. Strizhakova, and I.A. Sheka. Zinc ion effect on the electron-transfer process. A possible mechanism of synergism of antioxidants. *Zhurnal Obshch. Chim. (Russ.)*, 1980, **50**, No.3, pp. 702-703.
5. Y.A. Maletin, N.G. Strizhakova, and I.A. Sheka. "Radical-nucleophilic" splitting the Cu(II) diketonates with verdazyl radicals. *Zhurnal Obshch. Chim. (Russ.)*, 1981, **51**, No.5, pp. 1119-1126.
6. L.S. Degtyaryov, Y.A. Maletin, and A.A. Stetsenko. On the verdazyl radical disproportionation in the presence of zinc or cadmium ions. *Zhurnal Obshch. Chim. (Russ.)*, 1981, **51**, No.10, pp. 2387-2388.
7. Y.A. Maletin et al. Interaction of some Cu (II) chelates with nitroxyl or verdazyl radicals. *Coord. Chim. (Russ.)*, 1981, **7**, No.10, pp. 1464-1470.
8. Y.A. Maletin. Nature of chemical bonding in 3d-metal β -diketonates. In "Problems of Chemistry and Applications of Metal β -diketonates." Ed. V.I. Spitsyn (*Russ.*), Moscow, Nauka, 1982, pp. 5-11.
9. N.G. Strizhakova, Y.A. Maletin, and I.A. Sheka. Effect of metal complex formation on the verdazyl radical disproportionation. *Zhurnal Obshch. Chim. (Russ.)*, 1983, **53**, No.3, pp. 498-502.
10. Y.A. Maletin, V.N. Statsyuk, and N.G. Strizhakova. 3d-Metal ion effect on polarographic characteristics of some amines and radicals. *Ukrain. J. Chem. (Russ.)*, 1983, **49**, No. 9, pp. 996-997.
11. Y.A. Maletin, A.S. Tatikolov, and I.V. Khudyakov. Zinc ion induced dismutation of triphenylverdazyl radicals. *Oxidat. Commun.*, 1983, **5**, Nos. 3-4, pp. 415-422.
12. I.V. Khudyakov, Y.A. Maletin, and B.I. Yakobson. Effect of solvent viscosity and polarity on the rate of aromatic radical oxidation by copper (II) compounds. *Oxidat. Commun.*, 1984, **7**, Nos. 3-4, pp. 355-367.
13. I.A. Sheka, Y.A. Maletin, and N.G. Strizhakova. Nonadiabaticity of electron-transfer reactions via bridge metal-ion. *Doklady AN USSR, Ser.B (Russ.)*, 1984, No.6, pp. 48-50.
14. N.G. Strizhakova, Y.A. Maletin, and I.A. Sheka. Interaction of some 3d-metal β -diketonates with triphenylverdazyl radicals. In "Theoretical and Applied Chemistry of Metal β -diketonates." Ed. V.I. Spitsyn and L.I. Martynenko (*Russ.*), Moscow, Nauka, 1985, pp. 163-166.
15. Y.A. Maletin. Transition metal β -diketonates as inhibitors of oxidation processes. In "Theoretical and Applied Chemistry of Metal β -diketonates." Ed. V.I. Spitsyn and L.I. Martynenko (*Russ.*), Moscow, Nauka, 1985, pp. 256-262.
16. Y.A. Maletin et al. Electron-transfer reactions induced by zinc ions. *Ukrain. J. Chem. (Russ.)*, 1985, **51**, No. 2, pp. 148-151.
17. Y.A. Maletin, T.V. Verhovlyuk, and I.A. Sheka. Kinetics and mechanism of copper (II) dithiocarbamate formation in acetonitrile. *Ukrain. J. Chem. (Russ.)*, 1985, **51**, No. 8, pp. 787-791.

{WP269194;1}

18. Y.A. Maletin et al. Interaction between triphenylverdazyl radicals and copper, iron, aluminium or zinc complexes with 3-N-trifluoromethyl phenylanthranilic acid. *Coord.Chim. (Russ.)*, 1985, 13, No.12, pp. 1620-1625.
19. Y.A. Maletin, and N.G. Strizhakova. Kinetics and mechanism of electron transfer via bridge metal-ion. *Intern.J.Chem.Kinet.*, 1986, 18, No.1, pp. 13-29.
20. Y.A. Maletin et al. Electro-catalytic reduction of molecular oxygen in complex electrolytes. In "Electrocatalysis and Electrocatalytic Processes". (Russ.). Kiev, Naukova Dumka, 1986, pp. 29-44.
21. D.V. Matyushov, and Y.A. Maletin. Inner-sphere electron transfer accompanied by the rupture of metal-ligand bonding. *Doklady AN USSR, Ser.B (Russ.)*, 1988, No.2, pp. 52-55.
22. N.G. Strizhakova, Y.A. Maletin, and S.I. Travin. Autooxidation of copper (I) phenanthrolineates or dithiocarbamates in acetonitrile. *Coord.Chim. (Russ.)*, 1988, 14, No.6, pp. 738-743.
23. Y.A. Maletin et al. Mechanism of the ferrocene oxidation by iodonium salts. *Ukrain. J. Chem. (Russ.)*, 1988, 54, No. 8, pp. 816-818.
24. Y.A. Maletin et al. Kinetics and mechanism of the cuprous ion oxidation by thiuram disulphide. *Theoret. Experim. Chim. (Russ.)*, 1988, 24, No.4, pp. 450-455.
25. Y.A. Maletin, A.A. Dubinskaya and K.E. Gulyanitsky. Activation parameters of electron-transfer reactions with concerted bond rupture. *Theoret. Experim. Chim. (Russ.)*, 1988, 24, No.4, pp. 501-504.
26. D.V. Matyushov, and Y.A. Maletin. Electron transfer accompanied by bond rupture. *Chem. Phys.*, 1988, 127, Nos. 1-3, pp. 325-334.
27. O.A. Mashkin, Y.V. Kuzminskii, and Y.A. Maletin. Physicochemical techniques in the investigation of electrochemical cells. *J. Power Sources*, 1991, 34, No.1, pp. 175-182.
28. Y.A. Maletin et al. Specific character of solvation effects in the inner-sphere electron-transfer reactions. *Ukrain. J. Chem. (Russ.)*, 1991, 57, No. 5, pp. 451-454.
29. Y.A. Maletin. Classification of redox-reactions of complex compounds. *Ukrain. J. Chem. (Russ.)*, 1993, 59, No. 5, pp. 519-526.
30. Y.A. Maletin et al. Prospects of non-aqueous electrolyte applications in aluminium capacitors. *Ukrain. J. Chem. (Russ.)*, 1993, 59, No. 8, pp. 838-843.
31. Y.A. Maletin et al. Model for oxide film growth in aluminium anodization. *Theoret. Experim. Chim. (Russ.)*, 1994, 30, No.5, pp. 272-276.
32. Y.A. Maletin et al. Kinetics of electrode reduction of tetrachlorocuprate (II) ion: an example of the concerted EC reaction mechanism. *J. Electroanal. Chem.* 1995, 398, pp. 129-134.
33. Y.A. Maletin et al. Novel type of storage cells based on electrochemical double-layer capacitors. In "New Promising Electrochemical Systems for Rechargeable Batteries". Ed. V. Barsukov & F. Beck, NATO ASI Series, Kluwer, 1996, pp. 363-372.
34. Y.A. Maletin et al. Kinetics of oxide film growth in anodic oxidation of aluminium. *Theoret. Experim. Chim. (Russ.)*, 1997, 33, No.1, pp. 58-61.
35. Y.A. Maletin et al. A new type of binuclear oximehydrazonate clathrochelates of iron(II): synthesis, spectra and structure. *Inorg. Chim. Acta*, 1997, 255, pp.255-268.
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37. Y.A. Maletin et al. Correlation time of acetonitrile and its self-association in binary mixtures with non-polar solvents. *Ukrain. Chem. J., (Russ.)* 1997, 63, No. 10, pp. 102-106.

{WP269194;1}

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39. Y.A. Maletin, and R.D. Cannon. Dissociative electron transfer reactions. *Theoret. Experim. Chim., (Russ.)* 1998, **34**, No. 2, pp. 67-78.
40. Y.A. Maletin et al. Macrobicyclic iron(II) oximehydrazonates and α -dioximates formed by capping with antimony(V) triorganyles: the first synthesis of antimony-containing clathrochelates. *Inorg. Chem. Commun.*, 1998, **1**, pp. 328-331.
41. Y.A. Maletin, et al., Synthesis, spectral and electrochemical characteristics of asymmetrical iron(II) tris-dioximates. *Polyhedron*, 1998, **17**, No. 25-26, pp. 4315-4326.
42. Y.A. Maletin, et al., New capping agents for oximehydrazonate clathrochelates: sterically controlled synthesis, structural characterization and intramolecular reactions. *Inorg. Chim. Acta*, 1999, **284**, pp. 180-190.
43. Y.Y. Zhook, N.G. Strizhakova, and Y.A. Maletin. Ferrocene-containing ligands in a self-assembling reaction of μ_3 -oxocentered Cr(III,III,III) carboxylate complexes. *Theoret. Experim. Chim., (Russ.)* 2000, **36**, No. 4, pp. 233-237.
44. Y.A. Maletin, et al., Coordination compounds of N,N-dimethylhydrazides of aryloxycarbonic acids with some 3d-metal nitrates. *Coordin. Chim. (Russ.)*, 2000, **26**, No. 6, pp. 446-452.
45. Y.A. Maletin, et al., Synthesis and properties of trinuclear μ_3 -oxocentered manganese acetate complexes. *Ukrain. Chem. J. (Russ.)*, 2000, **66**, No. 6, pp. 72-76.
46. Y.A. Maletin, et al., Effect of heterosubstitution on the properties of trinuclear μ_3 -oxocentered carboxylate complexes of chromium, iron and manganese. *Ukrain. Chem. J. (Russ.)*, 2000, **66**, No. 8, pp. 76-81.
47. Y.A. Maletin, et al., Proc. 10th Intern. Seminar on Double Layer Capacitors and Similar Energy Storage Devices, Deerfield Beach, Florida, USA, Dec. 2000.
48. N.A. Kostromina, O.P. Kryatova, and Y.A. Maletin. Complexes of metals with boron-containing ligands. *Ukrain. Chem. J. (Russ.)*, 2000, **66**, No. 12, pp. 67-76.
49. Y.A. Maletin, et al., The synthesis and structure of nickel(II) and copper(II) complexes with N,N-dimethyl-N'-(2,4-dichlorophenoxy)acetyl hydrazine. *Inorg. Chem. Commun.*, 2001, **4**, 134-137.
50. Y.A. Maletin, et al., Lability or rigidity of the coordination sphere due to the change of the central atom oxidation state in 3d-metal complexes. *Ukrain. Chem. J. (Russ.)*, 2001, **67**, No. 7, pp. 32-36.
51. Y.A. Maletin, et al., Conductivity of concentrated solutions of onium salts in polar aprotic solvents. *Ukrain. Chem. J. (Russ.)*, 2001, **67**, No. 9, p. 6-8.
52. Y.A. Maletin, et al., Mass-spectroscopic study of gas phase reactions of macrobicyclic iron(II) complexes. *Ukrain. Chem. J. (Russ.)*, 2002, **68**, No. 2, p. 76-83.
53. Y.A. Maletin, et al., 3d-Metal complexes with N,N-dimethylhydrazide of furane-carbonic acid. *Ukrain. Chem. J. (Russ.)*, 2002, **68**, No. 4, p. 69-74.
54. Y. Maletin, et al. Simulation of Electrochemical Processes on the Nanoporous Carbon Material/Aprotic Electrolyte Interface, *Visnyk Lviv. Univ. (Ukrain.)*, 2002, Part 1, No. 42, p. 94-97.
55. Y.A. Maletin, et al., Proc. Intern. Conference on Advanced Capacitors, Kyoto, Japan, May 2003, p. 63-64.
56. Y.A. Maletin, et al., Proc. Int. Conference "Promising Electrochemical Systems for Batteries", Kiev, Ukraine, Sep. 2003, p. 26-27.
57. Y.A. Maletin, et al., Abstracts NATO Carbon Advanced Research Workshop and Conference, Argonne National Laboratory, USA, Oct. 2003, p. 18.

{WP269194;1}

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Patents

(NOTE: patents of the former Soviet Union, SU Pat., were closed for publication – this was the usual practice in the FSU)

1. Y.A. Maletin et al. Method of obtaining the "zineb" fungicide. SU Pat. 1 136 445, 1984.
2. Y.A. Maletin et al. Zinc N-methyl-N-tetrafluoropropyl dithiocarbamate as an antioxidant for lubricative oil. SU Pat. 1 330 991, 1987.
3. Y.A. Maletin et al. An electrolyte for fuel cell. SU Pat. 1 417 729, 1988.
4. Y.K. Delimarskii, Y.A. Maletin et al. Method of obtaining boron trichloride. SU Pat. 1 464 509, 1988.
5. Y.A. Maletin et al. Method for examining the quality of welded joints. SU Pat. 1 447 079, 1988.
6. Y.A. Maletin et al. Active mass of a positive electrode for electrochemical power cell. SU Pat. 1 470 133, 1988.
7. Y.A. Maletin et al. Method of obtaining nickel coating. SU Pat. 1 494 558, 1989.
8. A.V. Gorodyskii, Y.A. Maletin et al. Electrochemical power cell. SU Pat. 1 570 589, 1990.
9. Y.A. Maletin et al. Electrolyte for the electrochemical double-layer capacitor. SU Pat. 1 760 900, 1992.
10. Y.A. Maletin et al. Electrochemical accumulator based on a double-layer capacitor. PCT laid-open patent applied, PCT/RU94/00006, publ. WO 95/20231.
11. Y.A. Maletin et al. Tetrakis(N,N-dialkylamido)phosphonium tetrafluoroborates as electrolytes for double layer capacitors. RU 2022926, filed 930426, publ. 941115.
12. Y.A. Maletin et al. Tetrakis(N,N-dialkylamido)phosphonium tetrafluoroborates as electrolytes. UA 26919, filed 930705, publ. 991229.
13. Y.A. Maletin et al. Electrolyte for electrochemical double-layer capacitor. UA 21528 A, filed 950320, publ. 971216.
14. Y.A. Maletin et al. Tetrafluoroborates and hexafluorophosphates, their preparation and their use. UA 290624, filed 971225, publ. 001016.
15. Y.A. Maletin et al., Electrolyte for electrochemical double layer capacitor. UA 30509A, filed 980518, publ. 001115.
16. Y.A. Maletin et al. Electrolytes for electrochemical double layer capacitors. US 6,491,841; filed 990518, publ. 021210.
17. Y.A. Maletin et al. Method of making the electric double layer capacitor. UA 54508, filed 991117, publ. 030317.
18. Y.A. Maletin et al. Supercapacitor and a method of manufacturing such a supercapacitor. US 6,602,742, filed 001111; publ. 030805.
19. Y.A. Maletin et al. Supercapacitor and a method of manufacturing such a supercapacitor. US 6 697 249, filed 020923, publ. 040224.

{WP269194;1}